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UV/Ozone Cleaning of Silicon Carbide Optics

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Abstract

This document summarizes the work done to evaluate the effects of UV/Ozone cleaning on silicon carbide mirrors.

Introduction

UV/Ozone cleaning[1] was initially developed to clean semiconductor wafers and to assist in semiconductor device manufacture. More recently, it has been shown that UV/Ozone cleaning can be used to clean carbon contamination [2, 3] from synchrotron radiation optics. Although UV/Ozone cleaning has succeeded in cleaning synchrotron radiation optics, the method is very strongly oxidizing and can damage some optical surfaces. The method is well established for cleaning of stable surfaces like glass, fused silica, gold, and platinum. On the other hand, the method has notably failed and damaged optical surfaces of osmium[4, 5], nickel[6], and silver. In previous studies, UV/Ozone cleaning has been tested for use on replica gratings[7] and nickel optical surfaces [6]. It is the purpose of this work to evaluate UV/Ozone cleaning for use on silicon carbide optical surfaces. The properties of silicon

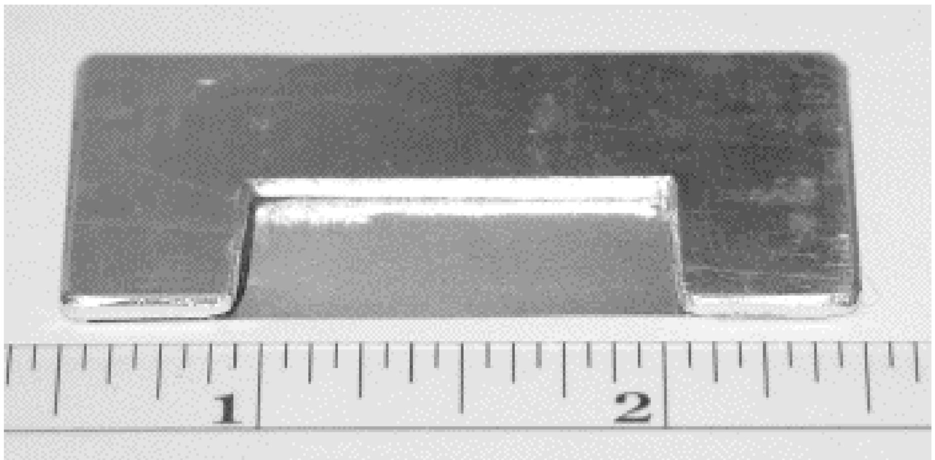

Figure 1: This is the inverted cleaning mask. It was designed to cover half of the optical surface of the test mirror during UV/Ozone cleaning.

carbide make it attractive for synchrotron radiation mirrors. The material is extremely hard and durable, and can be polished to an excellent surface finish. It also has good thermal conductivity and can be used for water cooled optics. The justification for this study was the medium energy grating (MEG) of the Wadsworth beamline at SRC. This grating is an ion etched grating made from silicon carbide. As received from the manufacturer, the high energy flux reflected by the medium energy grating was extremely low. It was speculated that this could be due to remnants of the photoresist used in manufacture, and there was a desire to clean the grating to remove any possible remnants of photoresist. UV/Ozone cleaning is effective in removing photoresist; however, prior to cleaning our grating, we wanted to perform tests to establish that the silicon carbide optical surface would not be degraded by the cleaning process.

Experimental

Two Silicon Carbide witness mirrors were ordered from Valley Design Corporation [8]. The mirrors were product number SC 103 with dimensions of 1" by 1" by 0.020". The mirrors were figured and polished on one side with 1/4 wave flatness. Mirror 1 was left as received, and mirror 2 was UV/Ozone

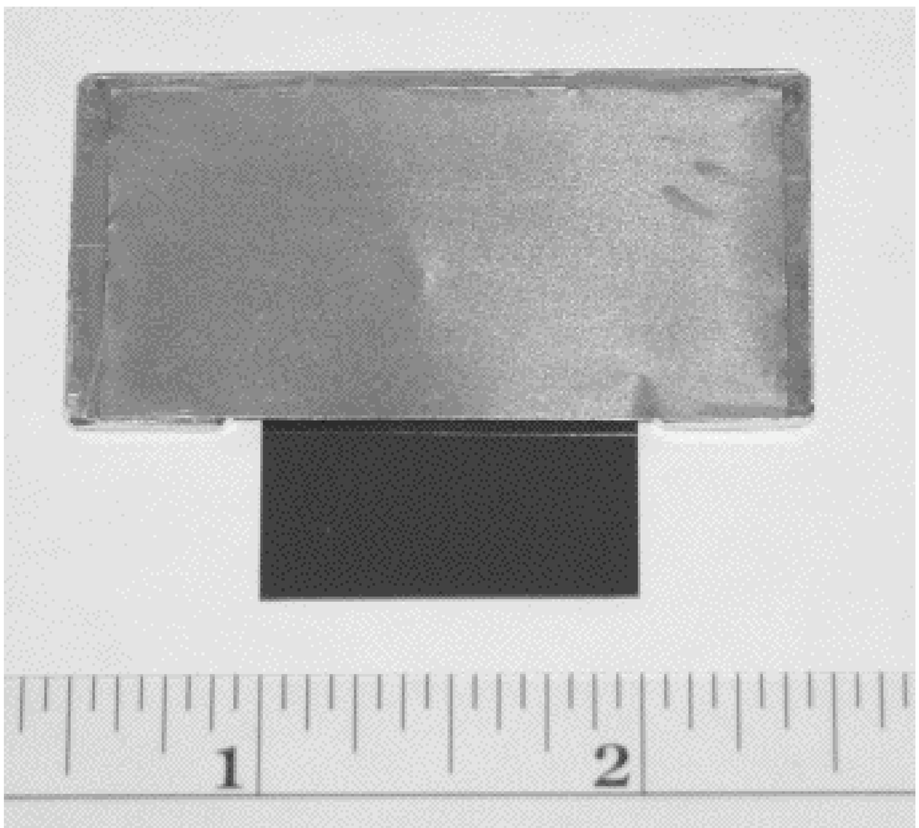

Figure 2: This shows the cleaning mask positioned over half of the mirror in preparation for UV/Ozone cleaning.

cleaned.

A mask, illustrated in figure 1, was made to cover half of the mirror during the cleaning process. The mask consisted of a thin piece of shim stock attached to a metal frame that fit around the outside of the mirror and held the shim stock about 0.040" above the mirror surface. The mask fitted over the mirror is shown in figure 2. With this mask in place during UV/Ozone cleaning, the uncovered half of the mirror was exposed to UV/Ozone cleaning conditions. The covered half of the mirror was exposed to some ozone but not to significant amounts of UV light. Generally, carbon oxidation occurs more readily in the unshielded area exposed to UV light and ozone. Nickel, in contrast is more readily degraded in the shaded area exposed to ozone alone [6]

The mirror and mask were centered under the cleaning lamp with a lamp to mask distance of about 5 mm. They were exposed to UV/Ozone cleaning for a period of 60 hours. This time period is considerably longer than normally required to remove synchrotron radiation contamination (about 7 hours). The mirror was deliberately over exposed to make any deleterious effects of cleaning more evident. The cleaner[9] consists of an acrylic desiccator with a volume of one cubic foot. The lamp is suspended from the top shelf, and the sample is placed below it on the next shelf. During the cleaning period, a small pump provides filtered air at a slow rate (about 2 liters per minute). The effluent from the cleaner goes through two columns of activated charcoal to neutralize the ozone before it is vented to the laboratory. The other mirror (mirror 1) was left as it was received by the manufacturer.

Table 1. Results of the roughness measurements on mirror 1.

Optic (#)	**Meas.** (#)	**5x objective** (Profile length: 2.66 mm 1016 valid data points)		**40x objective** (Profile length: 0.33 mm 1016 valid data points)	
		RMS (nm)	**P-V (nm)**	**RMS (nm)**	**P-V (nm)**
#1	1	9.8	46.1	4.4	18.4
	2	7.6	38.8	4.6	24.1
	3	8.0	36.4	6.2	33.3
	4	8.6	44.7	5.6	26.6
	5	7.3	33.3	6.0	41.8
	6	8.3	40.3	6.0	26.0
	7	7.8	39.7	4.7	23.1
	8	9.1	45.7	5.7	32.9
	9	8.3	50.1	7.3	31.1
	10	8.5	49.5	6.3	57.6
	11	7.3	33.2	5.5	30.1
	12	7.9	43.7	5.8	27.9
	Mean	8.2 $\pm$ 0.7	41.8 $\pm$ 5.8	5.7 $\pm$ 0.8	31.1 $\pm$ 10.1

Interferometer tests

After treatment of mirror 2, both mirrors were shipped to Argonne National Laboratory for testing with a TOPO2D roughness profiler [10]. This instrument uses the phase-shifting interferometry technique [11] to measure roughness of a one-dimensional surface profile over a small area, with 1 Ångstrom height resolution. The size of the probed area and the sampled spatial frequencies depend on the instrument objective lens. In this work, two different objective lenses were used: a 5x and 40x objective. The individual measured profiles are 2.66 mm and 0.33 mm in length, respectively, and the corresponding sampling distances are approximately 2.6 and 0.3 μm, respectively. Unfortunately, when they arrived, it was discovered that mirror 1 had come loose in shipping. When the package was opened, mirror 1 was lying on top of mirror 2. It is not possible to know at what point in transit the mirror came loose. The shape of the package made it possible for the back of mirror 1 to rub on the optical surface of mirror 2, and for the optical surface of mirror 1 to rub on the plastic packaging. Potential damage should have been minimized by the low mass of the mirrors and the extreme durability of silicon carbide. Because we were under a time constraint, and visible damage appeared minimal, we went ahead and tested the mirrors. The instrument measures the surface roughness of a small area of the mirror surface and quantifies the surface roughness for a range of spatial frequencies. Because of the small sampling area, it is necessary to sample a large number of locations to obtain a result representing the entire mirror surface. Table 1 shows the results of 12 samples of the surface of mirror 1. Measurements were performed with a 5X and 40X objectives (2.66 and 0.33 mm). Each objective has a different sampling area and responds to different spatial frequencies. Both the root mean square (RMS) and peak to valley (P-V) values are given in the table. The peak to valley values are determined by the lowest and highest points in the sampled area. As such, they represent only two points on the surface. Pits or ridges are undesirable on optical surfaces; however, they usually represent only a small part of the surface and are difficult to totally eliminate. A pitted mirror can perform well if most of the mirror surface is in good condition. Because of this, the RMS values were used to make comparisons. These values are expected to more closely characterize the overall surface and should be more relevant to the performance of the mirror. Mirror 1 represents the control group and should be representative of the mirror 2 surface before UV/Ozone treatment.

Table 2. Results of the roughness measurements on mirror 2.

Surface Treatment	**Meas.** (#)	**5x objective** (Profile length: 2.66 mm 1016 valid data points)		**40x objective** (Profile length: 0.33 mm 1016 valid data points)	
		RMS (nm)	**P-V (nm)**	**RMS (nm)**	**P-V (nm)**
(Exposed to Ozone only)	1	8.3	42.2	8.0	35.2
	2	8.7	70.4	4.5	26.9
	3	8.2	40.3	7.1	35.2
	4	8.6	59.9	7.1	29.9
	5	7.2	57.6	5.7	34.1
	6	6.4	41.4	5.6	28.9
	7	7.2	32.0	4.7	25.3
	8	7.3	50.3	5.2	21.0
	9	10.0	113.0	5.7	25.9
	10	6.7	35.9	6.6	36.5
	11	11.3	63.2	4.0	20.4
	12	6.8	28.9	6.8	27.8
	Mean	8.1 ± 1.4	52.9 ± 23.0	5.9 ± 1.2	28.9 ± 5.5
(Exposed to UV/Ozone)	1	8.0	44.1	3.4	16.0
	2	8.4	36.3	5.4	23.1
	3	8.8	45.3	6.0	26.1
	4	8.3	42.1	6.8	32.5
	5	7.0	36.7	4.4	25.6
	6	7.0	39.3	6.34	49.8
	7	7.5	35.9	5.9	30.5
	8	8.8	45.3	4.2	23.1
	9	8.6	41.8	5.7	28.9
	10	6.6	41.0	5.2	29.8
	11	9.9	48.3	5.2	27.0
	12	7.7	46.3	5.6	32.6
	Mean	8.1 ± 0.9	41.9 ± 4.2	5.3 ± 1.0	28.8 ± 8.1

Table 2 shows the results from mirror 2. Twelve locations were sampled on each half of mirror 2. Table 3 summarizes the mean values of the surface roughness for the important areas. The RMS surface roughness values suggest that all three mirror areas are identical. Although the mean values of roughness for the different areas vary, (8.2, 8.1, 8.1) the variations are small compared with the standard deviations. Application of a statistical t-test to differences in the mean values indicates less than 80 per cent confidence for a difference between the means. It is also noticeable that in three cases, the treated areas have a lower surface roughness than the control area. This would not be the result expected if UV/Ozone cleaning were damaging the optical surface. The possible damage to the mirrors in shipping is a serious flaw in this experiment. It is possible, that both mirrors have nearly the same RMS roughness, because they were both damaged by the same amount. Although it is not impossible, this is unlikely. I would have expected there to be visual evidence in this case, and I would have expected mirror 2 to be most seriously damaged. Mirror 2 having the same or slightly better surface roughness suggests little damage from the incident.

Table 3. Summary of the surface roughness measurements of the two mirrors.

Optic (#)	**Value**	**5x objective** (Profile length: 2.66 mm 1016 valid data points)		**40x objective** (Profile length: 0.33 mm 1016 valid data points)	
		RMS (nm)	**P-V (nm)**	**RMS (nm)**	**P-V (nm)**
#1	Mean	8.2 ± 0.7	41.8 ± 5.8	5.7 ± 0.8	31.1 ± 10.1
#2 Ozone only	Mean	8.1 ± 1.4	52.9 ± 23.0	5.9 ± 1.2	28.9 ± 5.5
#2 UV/Ozone	Mean	8.1 ± 0.9	41.9 ± 4.2	5.3 ± 1.0	28.8 ± 8.1

Conclusions

In conclusion, the surface roughness measurements indicate no significant degradation of the mirror surface finish caused by 60 hours of UV/Ozone cleaning. This strongly indicates that mirrors made from silicon carbide can

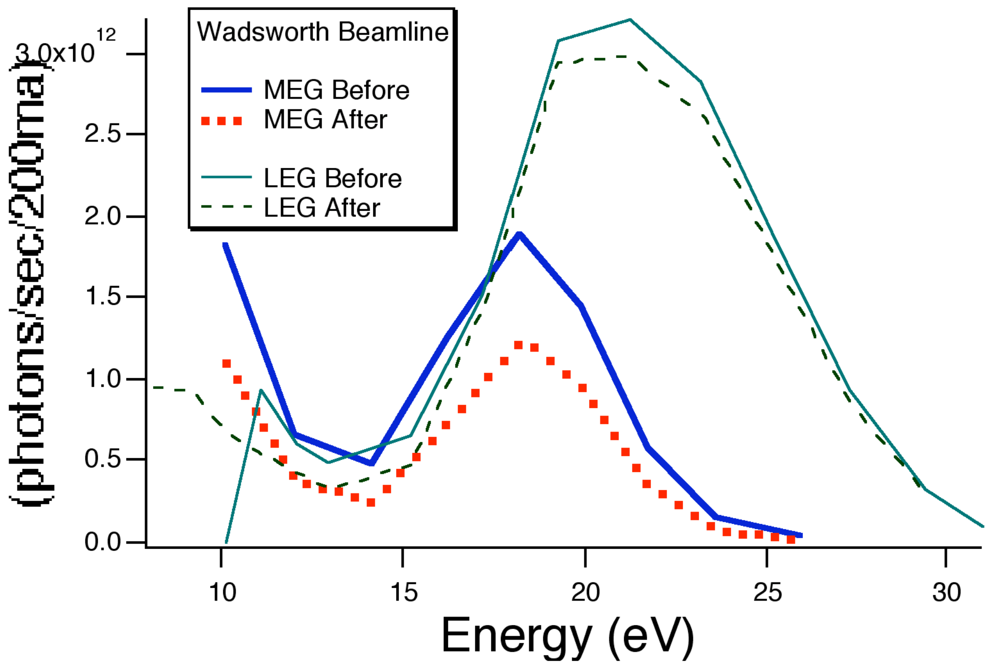

Figure 3: These are curves of the Wadsworth medium energy grating (MEG) flux before and after UV/Ozone cleaning and realignment. Flux values for the low energy grating (LEG) are also shown from before and after the MEG cleaning as a standard for the beamline.

be repeatedly cleaned to remove synchrotron radiation induced carbon contamination.

After these experiments showed no damage, the suspected Wadsworth monochromator medium energy grating was cleaned. During a maintenance period, the grating and its kinematic holder were removed from the monochromator and the grating was cleaned for 48 hours without demounting. The assembly was replaced in the monochromator and realigned. After restoration of the system, the performance of the grating was compared to performance before the cleaning process. Figure 3 shows the before and after flux measurements for the grating. There is a small decrease in flux after cleaning; however, there is also a decrease for the low energy grating which was not removed or treated in any way. It is more likely that these small changes are due to alignment, and other factors. There was no increase in medium energy grating flux at any point in the spectrum after cleaning. There was also no significant degradation of flux that could be blamed on the cleaning process. This supports our conclusion that silicon carbide is not vulnerable to attack by UV/Ozone cleaning and can be cleaned without difficulties. It also suggests that the low flux observed for the medium energy grating at higher energies is not caused by photoresist or other carbon contamination. We conclude that silicon carbide mirrors can be cleaned for reasonable cleaning times(10 hrs.) without degrading the optical surface.

Acknowledgment

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